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Superplasticity in Intermetallics(金属間化合物の超塑性)

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論 文 内 容 要 旨

In recent year, it has been shown that several single phase ordered intermetallic alloys with fine initial grain size such as Ni_3Al, $Ni_3(Si,Ti)$ and Co_3Ti can be deformed superplastically over 100% elongation at optimum deformation temperature or strain rate. DRX has been observed in $L1_2$ intermetallic alloys and in bcc derivative intermetallics such as Fe_3Si, Fe_3Al and $Fe_3(Al,Si)$ when they are deformed superplastically. Also, it has been found that the grain sizes of DRX strongly depend on Z (Zener-Hollomon parameter). Grain boundary sliding was suggested as a deformation mechanism responsible for superplastic deformation in the $L1_2$ single phase intermetallics with fine initial grain size. On the other hand, it has been suggested that superplastic deformation on the bcc derivative single phase intermetallics with coarse initial grain size is associated with DRX and dynamic recovery (DRV). There exist large differences on the deformation behavior between the $L1_2$ intermetallic alloys and the $D0_3$ intermetallic alloys although superplasticity occurs in both single phase intermetallics. Here, we have a question "why" superplastic deformation is possible in fcc derivative intermetallics with fine initial grain size, while superplasticity occurs in bcc derivative intermetallics with large initial grain size. To address this question single crystals as well as poly crystals with various grain size were used in this study. Superplastic deformation behavior in intermetallics has been investigated as functions of strain rate, temperature, crystal structure and grain size using B2 and $D0_3$-type Fe_3Si(Single and poly crystals), B2-type Fe_3Al, $L1_2$-type Co_3Ti intermetallic alloys. To clarify the microstructural changes correlated with deformation mechanism, OM, SEM, TEM and EBSP were used.

Chapter 1 presents research trends of superplasticity and proposed deformation mechanisms in order to provide the required background and purpose of this study.

Chapter 2 presents superplastic deformation of B2-type Fe_3Si (14at%Si) polycrystal. The emphasis is on the microstructural changes during superplastic deformation and flow behavior. Particular attention is paid to the DRX and DRV microstructure according to the deformation conditions on the specimens with large initial grain size.

Chapter 3 presents large elongation behavior of B2-type Fe_3Si (14at%Si) single crystal. Main purpose of this chapter is to clarify the deformation mechanism based on the mechanical and microstructural results obtained in chapter 2. The micro mechanism responsible for the large tensile elongation in the Fe_3Si single crystals was proposed. The comparable work hardening (by the glide motion of the dislocations) with the dynamic recovery (by the climb motion of the dislocations) takes place, and offers the steadily increased flow stress and moderate value of strain rate sensitivity, resulting in large plastic deformation and high necking resistance.

Chapter 4 presents superplastic deformation behavior of boron doped Fe-18at%Si alloy with $D0_3$ structure, which was investigated in tension at strain rates from 2.34×10^{-5} to 2.34×10^{-3} s^{-1} and at temperatures from 973 to 1223 K. The samples with a large initial grain size of 72 μm were H_2 gas-quenched immediately after high temperature deformation for microstructural observation and discussion of the mechanism of superplastic deformation. It is concluded from the microstructural observations that superplastic deformation of boron doped Fe-18at%Si is not associated with grain boundary sliding of DRX grains, but with repeated formation of relatively coarse DRX grains containing subgrain structures, and so retardation of cavity formation and growth.

Chapter 5 presents high temperature deformation behavior of Fe-18at%Si single crystal, which was investigated by tensile tests to further understand the superplastic deformation of coarse-grained Fe-18at%Si polycrystals. The dislocations activated in the specimen showing large tensile elongation are identified to be the <001>-type. Deformation microstructures leading to large tensile elongation consist of subgrains created by dislocation rearrangement due to active motion of glide and climb of dislocations. It is proposed that steady state-like flow due to dynamic recovery (by the glide and climb motion of dislocations) is responsible for the large tensile elongation.

Chapter 6 presents superplastic deformation of boron doped Fe-28at%Al with B2-type(at deformation temperature) having an initial grain size of about 100 μm which was investigated by tensile tests as a function of strain rate and testing temperature. Main purpose of this chapter is to study alloy system dependence on the deformation behavior with the same crystal structure and initial microstructure. Deformation microstructure corresponding to superplasticity is quite similar to that in chapter 2, but flow behavior leading to large elongation is slightly different, because it is strongly affected by dislocation structure which has been well explained as functions of temperature, composition, strain rate and also an amount of strain in this intermetallics.

Chapter 7 presents the superplastic deformation of $L1_2$-type Co_3Ti alloy, which was observed as functions of temperature, strain rate and initial grain size, and then characterized by the flow behavior, the constitutive equation and the deformation microstructure. In the region of higher strain rate (also lower temperature and larger initial grain size), the flow curve exhibits the stress peak followed by the rapid stress decrease, and the dynamic recrystallization (DRX) occurs. The activation energy of deformation in this region is estimated to be Q=158 kJ/mol, similar to that of the bulk diffusion. It is suggested that the mechanism associated with DRX is responsible for the deformation in this region. In the region of lower strain rate, (also higher temperature and smaller initial grain size), the flow curve exhibits the continuous work hardening until fracture, and the concomitant motion of grain boundary sliding and grain growth occurs. The activation energy of deformation in this region was estimated to be Q=80, kJ/mol suggestive of that of the grain boundary diffusion. Large tensile elongation, i.e. superplastic deformation beyond 200 % was observed in the latter region. It is suggested that the grain boundary sliding-based mechanism is responsible for the deformation in this region.

Chapter 8 presents the OIM(Orientation Imaging Microscopy) study during superplastic deformation of $D0_3$-type Fe_3Si intermetallics. Characteristic flow behavior, microstructure and microtexture using OIM are investigated in $D0_3$-type Fe_3Si intermetallic alloy at temperature of 1173 K and at strain rates of $3.3x10^{-3} \sim 3.3x10^{-5}$ s^{-1}. Three types of flow curve are exhibited and the flow curve responsible for superplastic deformation is in intermediate strain rate, that is, the flow curve exhibits steady state flow accompanied with initial work softening after a peak stress at a small strain. DRX(dynamic recrystallization) observed throughout the entire deformation conditions examined. At high strain rate($3.3x10^{-3}$ s^{-1}), refined DRX grains than prior grain size are observed, and grain boundary sliding between fine DRX grains occurs but superplasticity does not takes place. In the deformation condition leading to fine DRX grains, overall reduction of microtexture is observed with deformation. In contrast, no remarkable microtexture changes are appeared to the specimen showing superplasticity. On the basis of obtained results, superplastic deformation mechanism is suggested.

Chapter 9 presents the summary and conclusions of this study. The main coclusions are summarized as follows.

1. Superplasticity occurs in bcc derivative intermetallics with coarse grains, while it does in $L1_2$-type intermetallics with fine grains.
2. Grain boundary sliding is necessary in $L1_2$-type intermetallics because of high strength of grain interior.
3. Glide and climb motions of dislocations are needed to superplasticity in bcc derivative intermetallics. DRX is not always necessary for superplasticity.
4. No apparent change is observed in the texture of superplastically deformed bcc derivative intermetallics.

審査結果の要旨

難加工性材料である金属間化合物においては，初期複相組織を微細化することによって従来の金属や合金と同じように超塑性現象が発現することが知られている．しかしながら最近単相の金属間化合物でも超塑性が発現することが見出され注目を集めているがその変形機構は不明である．本論文は，単相の金属間化合物における超塑性変形機構を解明するために，代表的ないくつかの金属間化合物を選んで，単結晶試料と粗大および微細初期粒径を有する多結晶試料を作製し，超塑性変形に及ぼす結晶粒径，合金組成，結晶構造などの影響を系統的に調べるとともに，変形組織の形成過程を観察し，得られた結果に基づいて超塑性変形機構を議論したもので．全編 9 章よりなる．

第 1 章は序論であり，超塑性研究の現状と本研究の目的について述べている．

第 2 章では，B2 型 Fe_3Si (Fe-14mol%Si) 多結晶材の超塑性変形挙動について調べ，伸びの温度およびひずみ速度依存性は応力-ひずみ曲線の形状と変形組織に関連づけられることを明かにしている．また，超塑性変形により動的再結晶粒とサブグレインが形成されることを示している．

第 3 章では，Fe_3Si (Fe-14mol%Si)単結晶の超塑性変形挙動と変形組織について調べ，多結晶材と比較検討をすることにより，動的再結晶は超塑性のための必要条件ではないことを明らかにしている．

第 4 章では，$D0_3$型 Fe_3Si (Fe-18mol%Si) 多結晶材の超塑性変形挙動について調べ，超塑性伸びの特徴を明かにしている．

第 5 章では，$D0_3$型 Fe_3Si (Fe-18mol%Si) 単結晶材の超塑性変形挙動について調べ，第 4 章での多結晶材の結果と比較検討することにより，超塑性におけるサブグレイン組織形成の重要性を指摘している．

第 6 章では，B2 型 Fe_3Al 多結晶材の超塑性変形挙動について調べ，B2 型 Fe_3Si 多結晶材と同じ超塑性変形機構で説明できることを示している．

第 7 章では，$L1_2$型 Co_3Ti 多結晶材の超塑性変形挙動について調べ，Ni_3Al や $Ni_3(Si,Ti)$と同じように，動的再結晶と粒界すべりで超塑性が説明できることを明かにしている．

第 8 章では，$D0_3$型 Fe_3Si 多結晶材の超塑性変形における組織変化を Orientation Imaging Microscopy (OIM) により解析し，この新しい解析手法で得られる微細集合組織や隣接粒の方位関係などの情報が超塑性変形機構を議論するうえで不可欠であることを明らかにしている．

第 9 章は総括であり，本研究で得られた成果をまとめている．

以上要するに本論文は，結晶構造の異なる代表的な単相金属間化合物の超塑性変形挙動を調べるとともに，変形中に形成される微細組織を観察することにより，超塑性変形機構を明らかにしたものであり，超塑性現象の解釈と超塑性材料の応用に対して重要な知見を提供するもので，材料加工学の発展に寄与するところが少なくない．

よって，本論文は博士（工学）の学位論文として合格と認める．